(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 802 282 A1

(12) EUROPEAN PATENT APPLICATION

published in accordance with Art. 158(3) EPC

(43) Date of publication:
22.10.1997 Bulletin 1997/43

(21) Application number: 96935528.8

(22) Date of filing: 01.11.1996

(51) Int. Cl.[6]: D21H 17/26

(86) International application number:
PCT/JP96/03213

(87) International publication number:
WO 97/16597 (09.05.1997 Gazette 1997/20)

(84) Designated Contracting States:
DE FR NL SE

(30) Priority: 02.11.1995 JP 285479/95
02.11.1995 JP 285480/95

(71) Applicant: UNI-CHARM COMPANY LIMITED
Kawanoe-Shi, Ehime 799-01 (JP)

(72) Inventors:
- TAKEUCHI, Naohito
Uma-gun, Ehime 799-07 (JP)
- MONOBE, Masanori
Kakegawa-shi, Shizuoka 436 (JP)
- OKUDA, Toshiyuki
Mitoyo-gun, Kagawa 769-16 (JP)
- OKUBO, Toshiya
Kakegawa-shi, Shizuoka 436 (JP)

(74) Representative: Van Malderen, Joelle et al
Office Van Malderen,
Place Reine Fabiola 6/1
1083 Bruxelles (BE)

(54) PROCESS FOR PREPARING HYDROLYZABLE SHEET

(57) The invention relates to a process to improve wet tensile strength as well as water-swelling property of a water-disintegrable sheet to be used as a wet wipe for cleaning in household.

A mixture of water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having a degree of substitution = 0.30 - 0.60 and pH ≥ 5.0 and water-dispersible fibers is subjected to a foliating step of a process to obtain the water-disintegrable sheet and, before or after the foliating step of the process, the mixture is added with a base.

EP 0 802 282 A1

Printed by Rank Xerox (UK) Business Services
2.14.18/3.4

Description

FIELD OF THE INVENTION

The present invention relates to a process for manufacturing a water-disintegrable sheet adapted to be dispersed or dissolved in water.

BACKGROUND ART

Wet wipes for cleaning use in household etc. are well known. Water-dispersible or water-disintegrable sheet-like articles typically as wet wipes are also well known, which are rapidly dispersed or disintegrated in water so that they can be directly thrown away into water, for example, in a flush toilet, and flushed down a drain. It should be understood that terms used herein "water-dispersible" and "water-disintegrable" are synonymous with each other. Generally, wet wipes having such characteristics are required to have a high strength exhibited while they are used in a wet condition and also a rapid integration when they are thrown away into a large quantity of water. To assure that these strength and integration should be compatible with each other, Japanese Laid-open Patent Application No. Hei1-168999 discloses use of water-insoluble sodium/calcium salt of carboxymethyl cellulose or sodium salt of carboxymethyl cellulose. Japanese Patent Publication No. Sho48-27605 discloses a process for paper making wherein a wet paper sheet obtained from water-insoluble carboxymethyl cellulose is sprayed with aqueous solution of alkaline metal. Japanese Laid-Open Patent Application No. Hei3-167400 discloses a process for paper making including a step of mixing a water dispersion of paper making stuff with water-insoluble alkaline salt of carboxymethyl cellulose. Furthermore, Japanese Laid-Open Patent Application No. Hei5-25792 discloses a process for paper making wherein a paper sheet obtained from a mixture of water dispersion of paper making stuff and alkaline salt of carboxymethyl cellulose is impregnated with hydrated organic solvent containing multivalent metal ion.

Carboxymethyl cellulose and/or salt thereof conventionally used by the above-mentioned prior arts generally lose their initial water-insolubility, then show a tendency to swell and finally become water-soluble as degree of substitution (D.S.) and pH thereof increase. When these carboxymethyl cellulose and/or salt thereof are used as binders for paper making, the tendency to swell of the binders will make it difficult to get a uniform mixture of the binders with fibers dispersed in water. On the other hand, the binders having become water-soluble will be prohibited from being deposited on a sheet as an intermediate product, strength of the sheet will be not so improved as expected from an actual quantity of the binders mixed in a water dispersion of paper making stuff. Although an aqueous solution of the binders could be sprayed to a sheet obtained from paper making stuff, swollen carboxymethyl cellulose makes an aqueous solution thereof too viscous to be sprayed in a uniform condition.

SUMMARY OF THE INVENTION

In view of problems as has have been described above, it is a principal object of the invention to provide an improved manufacturing process for making a disintegrable sheet for wet wipes or the like to be used for cleaning sheet materials in household etc. wherein a good quantity of carboxymethyl cellulose and/or salt thereof can be efficiently deposited on the sheet.

The object set forth above is achieved, according to the invention, by a process for manufacturing a water-disintegrable sheet from carboxymethyl cellulose and/or salt thereof and water-dispersible fibers, said process being characterized in that a mixture of water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having a degree of substitution (D.S.) = 0.30 - 0.60 and pH ≧ 5.0 and water-dispersible fibers is subjected to a step of foliating to obtain said water-disintegrable sheet and, before or after said step of foliating of said process, said mixture is added with a base.

Said base is preferably sodium carbonate. The process according to the invention may further comprise a step of impregnating said water-disintegrable sheet with aqueous solution to obtain a wet wipe from said sheet.

DESCRIPTION OF PREFERRED EMBODIMENTS

Materials used in a manufacturing process of the present invention include water-dispersible fibers which can be foliated in the presence of carboxymethyl cellulose and/or salt thereof. While it is preferred to use pulp fiber, it is also possible to use natural fibers such as wool, regenerated fiber such as rayon fiber, semi-synthetic fiber such as acetate, or synthetic fiber such as nylon, polyester etc. For such fibers, low water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having D.S. = 0.30 - 0.60 and pH ≧ 5.0 is used as binders. In order to provide such binders with a high water-swelling property or a high water-solubility, a base is added to the binders before or after a foliating step of a sheet making process. The base can be added during the foliating step if needed. While any appropriate base and combination of bases can be applied, it is preferred to use sodium carbonate for the present invention. Preferably,

well known paper making techniques are used to foliate a mixture of fibers and binders. It is also possible to use a so-called wet or dry method for manufacturing nonwoven fabric or a method for manufacturing nonwoven fabric using water jet streams. The invention will be described more in detail.

Examples 1 - 5

Coniferous pulp for paper making (NBKP) and carboxymethyl cellulose and/or salt thereof were mixed together and dispersed in city water followed by adding a necessary quantity of sodium carbonate to the water dispersion for preparation of a paper making stuff. After the stuff had been settled for a time, it was subjected to a small-sized paper machine used exclusively for testing to obtain a wet paper sheet which was then dried at a temperature of 110°C for 90 seconds using a dryer of rotary drum type to obtain a dried sheet having a basis weight of 40g/m^2. Then, the dried sheet was sprayed and impregnated with aqueous solution of propylene glycol/calcium chloride/deionized water mixed together at a ratio of 30/0.5/69.5(by weight) so as to obtain a quantity corresponding to 2.5 times a weight of the dried sheets. The impregnated sheet was settled for 24 hours at a temperature of 20°C to obtain a wet wipe. Water-dispersibility and wet tensile strength of the wet wipe were tested under measuring conditions which will be described below. Test results for evaluating wet wipe examples prepared in accordance with the present invention (Example) and wet wipes for control (Control) are summarized in Table 1. Relationships between a series of Examples and a series of Controls are as follows:

(1) Example 1 and Control 1

The test results of Example 1 and Control 1 show effects of sodium carbonate (% by weight) added to carboxymethyl cellulose and/or salt thereof as measured in a dried state during a paper making process.

(2) Example 2 and Control 2

The test results of Example 2 and Control 2 show effects of a degree of substitution (D.S.) as well as pH of carboxymethyl cellulose and/or salt thereof.

(3) Example 3 and Control 3

The test results of Example 3 and Control 3 show effects of a total quantity (% by weight) of pulp and carboxymethyl cellulose and/or salt thereof.

(4) Example 4 and Control 4

The test results of Example 4 and Control 4 show effects of a period for which a water dispersion of pulp and carboxymethyl cellulose and/or salt thereof should be settled after an addition of sodium carbonate during a paper making process.

(5) Example 5 and Control 5

The test results of Example 5 and Control 5 show effects of a mixture ratio of pulp and carboxymethyl cellulose and/or salt thereof.

Evaluation of water-dispersion

A 10cm X 10cm sample sheet of wet wipe was immersed in 300ml of deionized water contained in a 300ml glass beaker followed by stirring by means of a magnetic stirrer(at 600rpm) and an extent of dispersion of the sample was observed. Observation results were classified as follows:

A: A sample sheet was disintegrated within 100 seconds.
B: A sample sheet was disintegrated within 200 seconds.
C: A sample sheet was not disintegrated within 200 seconds.

Evaluation of wet tensile strength

A sample sheet of wet wipe of 25mm wide and 150mm long was elongated at a chucking distance of 100m and at an elongation rate of 100mm/min to get tensile strength. It was found that tensile strength of a wet wipe of at least 300g

is sufficient for its practical use.

Results of the evaluations for Examples and Controls are shown by TABLE 1. From the results it is concluded that:

(1) carboxymethyl cellulose and/or salt thereof should present D.S. = 0.30 - 0.60 and pH ≥ 5.0 (Example 2); (2) a quantity of sodium carbonate should correspond to 10 - 400% by weight of carboxymethyl cellulose and/or salt thereof (Example 1); (3) a weight ratio of pulp : carboxymethyl cellulose and/or salt thereof should range from 98 : 2 to 55 : 45; (4) concentration of pulp and carboxymethyl cellulose and/or salt thereof in a paper making stuff should range from 0.5 to 5% by weight; and (5) a settling period after addition of sodium carbonate should be 30 minutes or longer.

[TABLE 1]

Test No.	Mix. ratio pulp/CMC	CMC		Pulp/CMC Concentration in stuff (%)	Quantity of Na_2CO_3 (% by weight of CMC)	Settling period for stuff (h)	Result of evaluation		Remarks
		D.S.	pH				Water-dispersion	Tensile strength (g/25mm width)	
Ex.1 (1)	87.5/12.5	0.43	6.1	1	80	2	B	466	
Ex.1 (2)	87.5/12.5	0.43	6.1	1	160	2	A	704	
Ex.1 (3)	87.5/12.5	0.43	6.1	1	400	2	B	737	
Control 1 (1)	87.5/12.5	0.43	6.1	1	2	2	C	448	
Control 1 (2)	87.5/12.5	0.43	6.1	1	0	2	C	326	
Ex.2 (1)	87.5/12.5	0.58	6.0	1	80	2	A	617	
Control 2 (1)	87.5/12.5	0.43	4.7	1	80	2	B	195	
Control 2 (2)	87.5/12.5	0.88	6.9	1	80	2	A	253	
Ex.3 (1)	87.5/12.5	0.43	6.1	3	160	2	A	589	
Control 3 (1)	87.5/12.5	0.43	6.1	0.04	160	2	A	271	
Control 3 (2)	87.5/12.5	0.43	6.1	10	160	2	-	-	impossible to foliate
Ex.4 (1)	87.5/12.5	0.43	6.1	2	160	1	B	466	
Control 4 (1)	87.5/12.5	0.43	6.1	2	160	0.16	C	428	
Ex.5 (1)	95/5	0.43	6.1	2	80	2	A	320	
Control 5 (1)	99.5/0.5	0.43	6.1	2	80	2	A	63	
Control 5 (2)	50/50	0.43	6.1	2	80	2	-	-	inseparable from dryer

(Note) CMC : carboxymethyl cellulose and/or salt thereof

Examples 6 - 7

Coniferous pulp for paper making (NBKP) and carboxymethyl cellulose and/or salt thereof were mixed together and

dispersed in city water to obtain a paper making stuff which was foliated by a small-sized paper machine used exclusively for testing. A wet sheet thus obtained was sprayed and impregnated with a quantity of aqueous solution of sodium carbonate corresponding to 2.4 - 8% by weight of carboxymethyl cellulose and/or salt thereof wherein said quantity of aqueous solution corresponded to 2.5 times a weight of the sheet as measured in its dry condition. Then, the sheet was dried at a temperature of 110°C for 90 seconds using a rotary drum type dryer and thereby a dried sheet having a basis weight of 40g/m^2 was obtained. The dried sheet was sprayed and impregnated with aqueous liquid consisting of propylene glycol/calcium chloride/deionized water mixed together at a ratio of 30/0.5/69.5(by weight). The impregnated sheet was settled for 24 hours at a temperature of 20°C to obtain a wet wipe. Extent of water-dispersion as well as wet tensile strength of the wet wipe were evaluated under the same condition as Examples 1 - 5. Relationships between Examples and Controls are as follows:

(1) Example 6 and Control 6

The test results of Example 6 and Control 6 show effects of a quantity of sodium carbonate (% by weight) added to carboxymethyl cellulose and/or salt thereof as measured in a dried state during a paper making process.

(2) Example 7 and Control 7

The test results of Example 7 and Control 7 show effects of degree of substitution (D.S.) as well as pH of carboxymethyl cellulose and/or salt thereof.

Result of the evaluations for the respective Examples and Controls are shown by TABLE 2.

[TABLE 2]

Test No.	Mix. ratio pulp/ CMC	CMC		Quantity of Na_2CO_3 (% by weight of CMC)	Result of evaluation	
		D.S.	pH		Water-dispersion	Tensile strength (g/25mm width)
EX.1 (1)	87.5/12.5	* 0.50	7.3	2.4	A	475
Ex.1 (2)	87.5/12.5	* 0.50	7.3	4.0	A	416
Ex.1 (3)	87.5/12.5	* 0.50	7.3	8.0	A	380
Control 1 (1)	87.5/12.5	* 0.50	7.3	0.0	C	438
Ex.2 (1)	87.5/12.5	* 0.43	6.1	8.0	A	401
Ex.2 (2)	87.5/12.5	* 0.50	7.3	8.0	A	380
Control 2 (1)	87.5/12.5	* * 0.64	3.9	8.0	A	133
Control 2 (2)	87.5/12.5	* 0.88	6.9	8.0	A	83

(Note)
1. CMC : carboxymethyl cellulose and/or salt thereof
2. * : sodium carboxymethyl cellulose
3. * * : acid type carboxymethyl cellulose

Effect of the Invention

According to the process of the present invention, water-dispersible fibers are mixed with binders comprising carboxymethyl cellulose and/or salt thereof having low swelling tendency or water-insolubility so that a large quantity of binders can be deposited on a sheet obtained from the mixture of the fibers and the binders. Such binders are denaturalized by addition of a base to water-swelling or water-soluble ones. These binders contribute to improvement of tensile strength of the sheet. And the sheet is thrown away into a large quantity of water after use, the binders can be rapidly dissolved in the water, allowing the sheet to be rapidly disintegrated and dispersed in the water.

Claims

1. A process for manufacturing a water-disintegrable sheet from carboxymethyl cellulose and/or salt thereof and water-dispersible fibers, said process being characterized in that:

   a mixture of water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having a degree of substitution = 0.30 - 0.60 and pH ≧ 5.0 and water-dispersible fibers is subjected to a foliating step to obtain said water-disintegrable sheet and, before or after said foliating step of the process, said mixture is added with a base.

2. The process according to Claim 1, wherein said base is sodium carbonate.

3. The process according to Claim 1 or 2, further comprising a step of impregnating said water-disintegrable sheet with an aqueous solution to obtain a wet wipe from said sheet.

4. The process for manufacturing a water-disintegrable sheet from carboxymethyl cellulose and/or salt thereof, said process being characterized by that:

   a mixture of water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having a degree of substitution = 0.3 - 0.60 and pH ≧ 5.0, water-dispersible fibers and a base is foliated to obtain said water-disintegrable sheet.

5. The process according to Claim 4, wherein said base is sodium carbonate.

6. The process according to Claim 4 or 5, further comprising a step of impregnating said water-disintegrable sheet with an aqueous solution to obtain a wet wipe from said sheet.

7. The process for making a water-disintegrable sheet from carboxymethyl cellulose and/or salt thereof and water-dispersible fibers, said process being characterized in that:

   a sheet foliated from a mixture of water-swelling or water-insoluble carboxymethyl cellulose and/or salt thereof having a degree of substitution = 0.30 - 0.60 and pH ≧ 5.0 and water-dispersible fibers is added with a base to obtain said water-disintegrable sheet.

8. The process according to Claim 7, wherein said base is sodium carbonate.

9. The process according to Claim 7 or 8, further comprising a step of impregnating said water-disintegrable sheet with an aqueous solution to obtain a wet wipe from said sheet.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP96/03213

A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl^6 D21H17/26

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int. Cl^6 D21H17/26

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP, 6-192991, A (Nichirin Kagaku Kogyo K.K.), July 12, 1994 (12. 07. 94)(Family: none)	1 - 6
Y	JP, 3-167400, A (Nichirin Kagaku Kogyo K.K.), July 19, 1991 (19. 07. 91)(Family: none)	1 - 9
Y	JP, 3-8897, A (Lion Corp.), January 16, 1991 (16. 01. 91)(Family: none)	7 - 9
Y	JP, 1-168999, A (Kao Corp.), July 4, 1989 (04. 07. 89)(Family: none)	3, 6, 9

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
January 6, 1997 (06. 01. 97)	January 14, 1997 (14. 01. 97)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (July 1992)